

reaction was stirred for 3½ hours at 78° C. 250 g water was added and the mixture cooled to 27° C. Acetic acid (12.9 g) was added to reduce the pH from 12.24 to 8.74.

The resulting mass was filtered giving a calcium D,L-tartrate filter cake (for recycle) and a clear filtrate. This filtrate was added to a solution of sodium bicarbonate (2.1 g), sodium carbonate (25.97 g) and water (90 g) at 55° C. The mixture was heated to 75° C. for one hour and then at 85° C. for an additional hour to precipitate calcium carbonate. The mixture was filtered hot giving a calcium carbonate wet cake and a filtrate. The filtrate contained the desired HOPTC and DOOHC products and had the following analysis:

TABLE V

COMPONENT	WEIGHT
HOPTC	22.09
DOOHC	5.38
Disodium D,L-tartrate	0.99
Disodium maleate	1.71
Disodium fumarate	1.70
Disodium malate	0.20

The reaction described above was repeated four more times, each time using the two filter cakes (the "D,L-tartrate cake" and the "carbonate cake") obtained from the previous reaction and recycled to the next synthesis reaction. In each case similar results were obtained. In all cases the synthesis reactions were run for three hours. The first hour of the reaction the temperature was about 80° C. and then during the last two hours it was held at 90° C. The desired product from each synthesis reaction after calcium D,L-tartrate removal was analyzed to determine its HOPTC and DOOHC content. The conversion efficiency of maleate was also calculated. The results of these analysis are presented in Table VI below.

TABLE VI

	First Reaction	First Recycle	Second Recycle	Third Recycle	Fourth Recycle
Initial Solids % in reaction	34.0	28.0	28.0	43.0	45.0
Final Solids % in reaction	72.0	71.0	67.0	67.0	68.0
Total	72.5	71.6	68.0	67.0	69.8
HOPTC + DOOHC %					
Ratio of HOPTC/DOOHC	4.1	4.7	5.4	4.5	5.6
Maleate Conv. %	78.0	75.0	73.0	74.0	76.0

This example demonstrates that calcium carbonate wet cake prepared under synthesis conditions can be recycled into the synthesis reaction via calcium maleate formation without loss of conversion efficiency.

The foregoing description is given for clarity of understanding only and no unnecessary limitations should be understood therefrom, as modifications will be obvious to those skilled in the art.

I claim:

1. A process for preparing a mixture of the alkali metal salts of 1-hydroxy-3-oxa-1,2,4,5-pentane tetracarboxylic acid and 3,6-dioxo-1,2,4,5,7,8-octane hexacarboxylic acid by means of a calcium ion catalyzed reaction which comprises:

- reacting the salts of maleic and D,L-tartaric acids in alkaline medium;
- reducing the calcium content of said reaction product mixture to the extent that the molar ratio of calcium to the D,L-tartrate succinate reaction

products is less than about 1:10 by precipitation of calcium carbonate;

c. recovering the calcium carbonate removed in step (b) and recycling it to step (a) to prepare additional amounts of reaction product, and

d. recovering and purifying the remainder of the reaction product from step (b).

2. A process of claim 1 wherein the calcium is removed from the reaction product in step b at a temperature of from about 85° C. to about 105° C.

3. A process of claim 1 wherein the calcium carbonate is formed in the reaction mixture by reaction with an alkali metal carbonate.

4. A process of claim 2 wherein the calcium carbonate is formed by reaction with an alkali metal bicarbonate.

5. A process of claim 3 wherein the alkali metal is sodium.

6. A process of claim 4 wherein the alkali metal is sodium.

7. A process of claim 1 further including the step of reacting the calcium carbonate recovered in step (c) with maleic acid to form a salt before recycle to step (a).

8. A process of claim 7 wherein the calcium maleate is added to the reaction mixtures of step (a) in the form of an aqueous slurry.

9. A process of claim 7 wherein the calcium carbonate is formed by reaction of calcium ions with carbon dioxide.

10. A process of claim 1 wherein the reaction medium of step (b) is maintained at a pH in the range of from about 10 to about 12.

11. A process of claim 1 wherein the mole ratio of carbonate to calcium in step (b) is 1.3:1.

12. A process of claim 1 wherein the solids content of the reaction mixture of step (a) is in the range of from

55% to about 60% by weight.

13. A process for preparing a mixture of the alkali metal of 1-hydroxy-3-oxa-1,2,4,5-pentane tetracarboxylic acid and 3,6-dioxo-1,2,4,5,7,8-octane hexacarboxylic acid which comprises the steps of:

a. forming an aqueous reaction mixture comprising from about 20% to 60% by weight of both calcium and monovalent cation salts of maleic acid and D,L-tartaric acid, said mixture corresponding to the over-neutralized mixture which is formed by combining:

(i) maleic and D,L-tartaric acids in a maleic to D,L-tartaric molar ratio of from about 0.5:1 to about 8:1;

(ii) a source of calcium cations in an amount such that the molar ratio of calcium to D,L-tartaric acid ranges from about 0.1:1 to 2.0:1 with the ratio of moles of calcium to total moles of maleic and D,L-tartaric acid being less than 1; and